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Effects of Metal Ions Present in Recycled Water on Washing of Reactive Dyed Cotton Fabric

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Abstract

Textile industry is a key source of contamination of heavy metal ions in water. These heavy metal ions include lead, cadmium, copper, arsenic, nickel, chromium, zinc, and mercury. These metals have been recognized as hazardous heavy metals because of their high degree of toxicity. Initially, copper was selected to determine the effects of metals present in recycled water during the washing process because of its extensive usage in the textile industry. However, copper becomes a part of process water while manufacturing and even a part of recycled water. Furthermore, recycled water contains such traces of metal that can cause problems during its reuse. In the current research, the presence of copper ions in water was simulated with copper sulphate and the obtained water was used for washing of fabric after reactive dyeing. The effects of salt on washing of three different types of reactive dyes, that is, monochlorotriazine, dichlorotriazine and vinylsulphone were evaluated by washing of dyed fabric. Moreover, evaluation process was achieved with different concentrations of salt at different temperatures, pH values, and time intervals. Besides, K/S, fixation%, color uniformity, color change, washing fastness, and rubbing fastness of samples were measured during evaluation. Results were also compared with detergent and water wash. Eventually, the results showed that copper has a prominent effect on fabric. Hence, it would be possible to allow certain concentrations of copper during the process of washing.

Index Terms: cotton, exhaust dyeing, metal ions, reactive dye, recycled water.

I. Introduction

World's fresh water reserve is finite and only 0.03% of the world's water reserves are available for human use. Anyhow, rapid population and industrial growth have increased the demand of water. Moreover,

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inappropriate water management systems have restricted the supply of available resources, while their demand remains constant [1]. Further, self-purification abilities also seem to be insufficient [2]. Countries suffering from water crisis or those likely to suffer, must conserve water, maintain supply and demand balance, pollute less, and reduce the environmental impacts of growing population [3]. The textile industry is one of largest contributors in water pollution [4], [5], and it consumes huge volumes of water in its varied processing operations. However, the amount of water required for textile processing largely varies. It further depends on the chemicals available, working conditions and site, the required quantity and quality of the fabric, processes implemented, and the water sources [6], [7]. The consumption of water is comparatively less in mechanical processes such as spinning and weaving than in textile, where water is consumed extensively in wet processing. In the current study, the application of almost all dyes, auxiliaries, and finishing chemicals to textile substrates is from water baths. The maximum consumption of water in 'textile wet processing' is as a solvent and washing or rinsing media. Besides, a huge amount of water is also consumed in the boiler, cooling water, steam drying, and for cleaning purposes [8]. However, the reduced sources of fresh water, increased demand and cost of water, and effluents have forced the textile industry to consider water conservation. [8] Moreover, waters have become more and more stringent because of the regular discharge of waste from the industry [9]. The presence of heavy metals has made water pollution more severe. During the last few decades, incremental concentrations of metals present in effluents have attracted considerable attention of ecologists, globally. Such concentrations have also begun to cause new concerns in most of the major metropolitan cities. Untreated, as well as apparently treated, industrial effluents contain variable amounts of heavy metals. Such metals include arsenic, lead, nickel, cadmium, copper, mercury, zinc, and chromium. In addition to it, these metals have adverse effects on the land as well as on aquatic systems. The presence of these metal ions in textile effluents has been reported by various publications. Further, various metal salts are used to precipitate the dye molecules in waste water [1], [10]-[12], while some are used for mordanting [13]-[15], such as copper salts [16]. Copper is also used as catalyst for 'waste water treatment' [17]. These metals are present in effluents [4], [5] even after recycling treatments in lower concentrations. Moreover, many cases are reported on the removal of color, COD, turbidity, but no

treatments are reported for the removal of metal ions from water during a process of recycling [18]-[20]. The number of approaches has been carried out for the potential reuse of recycled water in various procedures including dyeing. For some cases, this use was found to be more beneficial for various procedures, even with dyeing at lower shades [20]-[23]. However, after washing, there are some problems, such as reddish color on the dyed fabric. Thus, it is worth studying the effects of metal ions in washing processes. Copper is largely used for mordanting process and it is also present in waste water coming from various textile processing steps. So, in the current research, copper ions were used as a target to evaluate the effects of metal ions on washing them with reactive dyeing of cotton fabric. Hence, possible faith of treated or untreated textile effluents contain metal ions.

II. Materials and Method

A. Materials

Commercially available, fully scoured and bleached, plain woven, 100% cotton fabric with average weight 200 grams per meter square (GSM) was utilized in this particular study. The thread density of fabric was 84 ends per inch (EPI) and 62 picks per inch (PPI) with the warp count 20 weft count 18 Ne on average. Moreover, samples of equal weight of 3grams were prepared for all further processing. The three types of reactive dyes used in the current study, were Dichlorotriazine dye (CI Reactive Red 2), Monochlorotriazine dye (CI Reactive Orange 5), and Vinylsulphone dye (CI Reactive Blue 19). These were bought from Shanghai Chemical Company, China. All three dyes were of commercial grade and were immediately used as received. All other reagents, namely as sodium carbonate, ferric Sulphate, aluminium chloride, copper sulphate, chromium chloride, acetic acid, and sodium chloride, were commonly used as a laboratory reagent grade.

B. Dyeing

Dyeing was carried out by using the three types of reactive CI Reactive Red 2, CI Reactive Orange 5, and CI Reactive Blue 19 of 3% o.w.f in tap water with a liquor ratio 50:1. For all cases fabric samples were added into dye bath and after 10 to 15 minutes, salt was further added into dye bath in two portions. For reactive red 2, the highest temperature used was 40°C, but for orange 5 and blue 19 after 10 to 15 minutes of salt addition temperature

was raised up to 80°C and 60°C with increment rate of 1-2°C/min. Moreover, right after 20 minutes of obtaining the maximum temperature, sodium carbonate was further added to it and temperature was maintained at this level for 45 minutes for Dichlorotriazine and Monochlorotriazine dyes. Furthermore, 30 minutes were kept for Vinylsulphone dye and then it was further followed up by rinsing it with cold water and performing the drying procedure. Exhaustion percentages of sample were measured for all samples by measuring the absorption of dye bath right before and after the dyeing procedure. Total 29 samples were dyed to complete each dye procedure. Concentration of salt and alkali for red and orange dyes were 40 g/l and 10 g/l, but in case of blue 45 it was 15 mg/l.

C. Washing

After the completion of dyeing process, each dyed sample was washed with different concentrations of salt solution, temperature, pH values, and time intervals. To check the effect of different washing conditions one out of four factors (concentration, temperature, time, and pH) was varied. While the other three factors were kept constant. However, initially samples were washed at various concentration levels with detergent and water (sample 1-10), at constant temperature, time, and pH level. In this case, the concentration with best results was chosen. After that all samples were washed with selected concentration for optimization of temperature at constant time and pH (sample 11-15). Then temperature with the best results was selected and kept constant. Similarly, the best time intervals (sample 16-21) and pH level (22-29), were selected. This process was repeated for all dyes. Different ranges of concentration, temperature, time, and pH applied for washing process are given in table below.

D. Testing

1) Measurement of Color Uniformity:

Color uniformity throughout the samples is one of the most important factors during the process of dyeing. The standard deviation was used to calculate the color uniformity of the dyed samples by measuring K/S values on 20 random spots at maximum absorption wave length λ by using the following equations.

$$(\sigma) = \sqrt{\frac{\sum_{i=1}^n \left[\left(\frac{k}{s} \right)_{i\lambda} - \left(\frac{\bar{k}}{\bar{s}} \right) \right]^2}{n-1}} \quad (1)$$

$$\left(\frac{k}{s}\right)_\lambda = \frac{1}{n} \sum_{i=0}^n \left(\frac{k}{s}\right)_{i\lambda} \quad (2)$$

Where (σ) is the standard deviation, n is the number of measured spots, and (K/S)_{iλ} is the K/S value of each random spot.

2) Dye fixation%:

Dye fixation is generally determined as an estimate of the average proportion of dye which is actually fixed on a textile fiber. The lower fixation levels of reactive dyes are essential due to unavoidable dye hydrolysis during the process of dyeing. There have been various analytical ways for estimating the extent of dye fixation and dye hydrolysis. Today, the percentage of dye fixation is usually determined by using absorbance measurements of dye bath solution and/or color strength measurements of the fabric during dyeing.

$$F\% = \frac{K/S_2}{K/S_1} \times 100 \quad (3)$$

Where, K/S1 and K/S2 are K/S value before and after washing respectively.

3) Color Change (Lab values):

L*a*b* values (CIE L*a*b*) were measured before and after washing procedure to check the change which was further occurred due to washing by using data color machine. The total metric color difference between two samples, ΔECIELAB, is calculated as the square root of the sum of the squares of the three component differences[24], [25]:

$$\Delta E_{CIELAB} = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2} \quad (4)$$

Where, ΔL = L2(after wash) – L1(before wash); Δa = a2(after wash) – a1(before wash); Δb = b2(after wash)– b1(before wash)

4) Measurement of Color Fastness to Washing and Rubbing:

Color fastness to washing and rubbing were measured according to ISO 105-C06 and [ISO 105-X12:2001](#) respectively.

Results and Discussion:

E. Color Uniformity

1) Effect of Concentration:

Figure 1 represents that how the concentration of copper salt effects the color uniformity of dyed samples. Although, concentration does not show any distinct effect on the color uniformity, but the color uniformity is superior to other two dyes only due to red dye. For red dye, an increase in the salt concentration resulted in the decrease of color uniformity up to

concentration of 5mg/l and after that it decreased again. Maximum color uniformity was obtained at salt concentration of 0.2mg/l. Orange dye has shown different behavior than red dye. Furthermore, the standard deviation tended to decrease very slightly with the increase in salt concentration up to 10mg/l and then it further started increasing. Hence, the slight increase in color uniformity with increase in concentration. No significant effect was observed for blue dye, although color uniformity was found to be at lower levels at moderate concentration levels. Copper gave better results than water and detergent wash in case of red dye, but it was found to be higher in cases of orange and blue dyes. This has happened because copper sulfate dissolves exothermically in water and gives an aquo complex $[\text{Cu}(\text{H}_2\text{O})_6]^{2+}$, which possess octahedral molecular geometry and is paramagnetic. Copper ions make complex bonds with the dyes and settle down if the particle size is adjustable. If particle size is not larger enough, then either it stays in the solution or entraps in fiber structure, resulting in the increase or decrease in color uniformity.

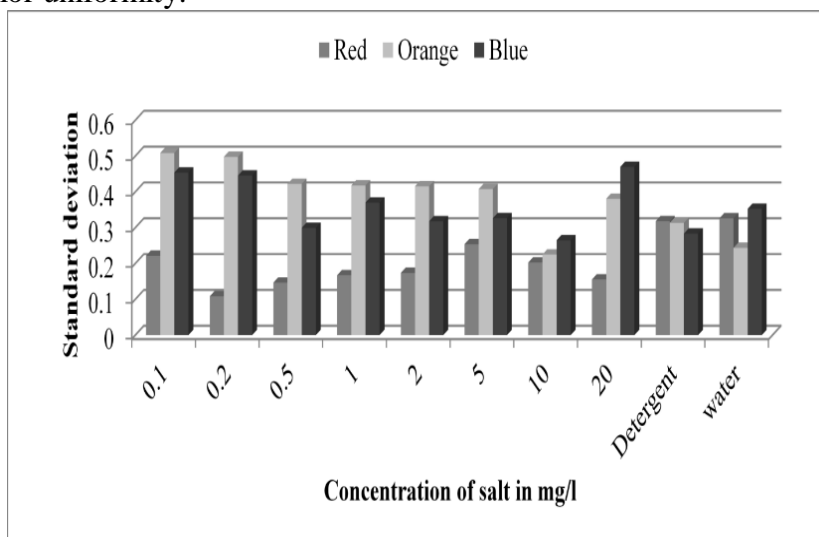


Fig. 1. Effect of salt concentration on color uniformity of samples washed with copper salt

2) Effect of Temperature:

Temperature had distinct effect on color uniformity as its values increased with increase in temperature. The standard deviation continued to decrease till 100°C for blue (vinylsulphone) dye, but for red (dichlorotriazine) and orange (monochlorotriazine) dye, it started increasing after 80°C and 60°C.

Since, the maximum color uniformity were obtained at 80°C, 60°C, and 100°C for red, orange, and blue dyes. Thus, it can be attributed to the phenomena that reactivity increases with the increase in temperature up to certain limits. Besides, kinetic energy of constituent's increases with the increase in temperature. Dye ions which are loosely attached with fabric or present on the surface of fabric, are more eager to react with metal particles. Hence, resulting in an increase among overall color uniformity. Effect of temperature on color uniformity can be seen in Fig. 2.

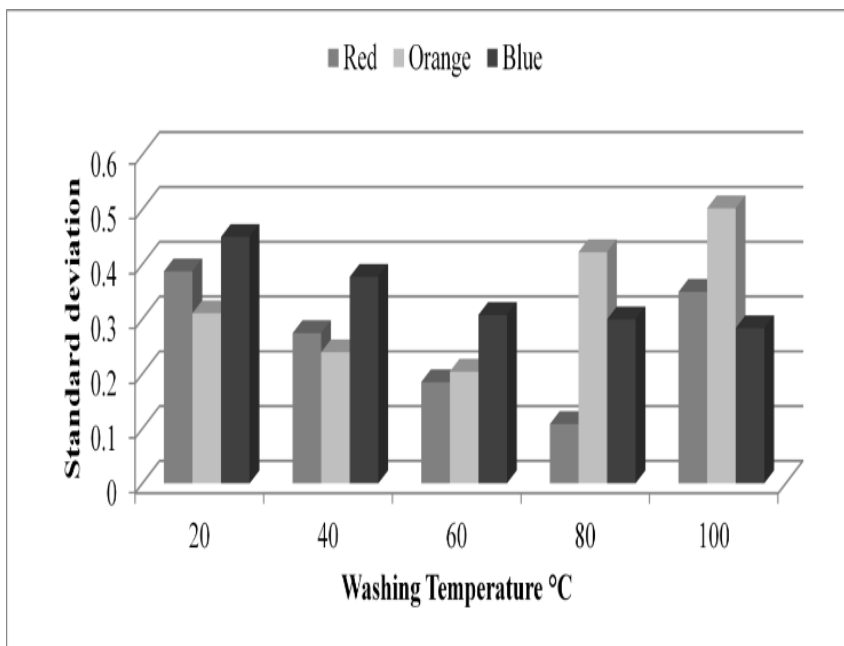


Fig. 2. Effect of washing temperature on color uniformity of samples washed with copper salt

3) *Effect of Time:*

Washing time had a positive effect on the color uniformity as it increased with the increase in washing time shown in Figure 3. The maximum color uniformity was obtained for red dye at 60 min washing time, but the minimum values of color uniformity were obtained at 40 and 50 min for orange and blue dyes. Furthermore, right after this calculation the standard deviation tended to increase. This increase in color uniformity up to certain limit is maybe due to the availability of longer reaction time. In addition to it, this longer reaction time resulted in increased color

uniformity for water and unfixed dye. ‘Longer reaction times’ means more chances of collision or interaction, and also the increased solubility of participants.

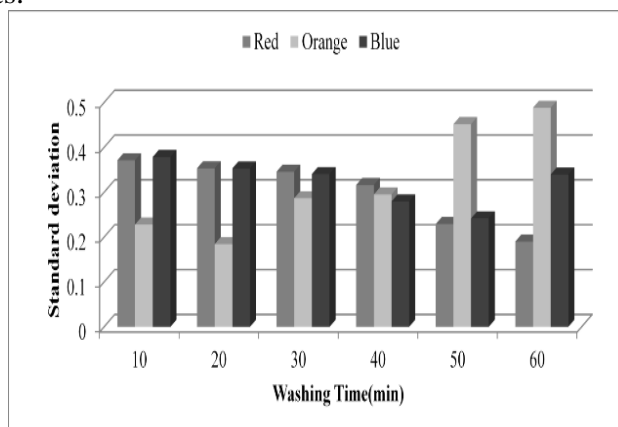


Fig. 3. Effect of washing time on color uniformity of samples washed with copper salt

4) Effect of pH:

However, lower values of color uniformity were obtained at moderate or slightly acidic or basic pH levels. Highly acidic pH levels were found to have worst effects on the color uniformity. Although, 10 all dyes showed higher values of standard deviation only at their pH level. While at other pH levels, color uniformity values were found to be optimum and approximately constant (Figure 4).

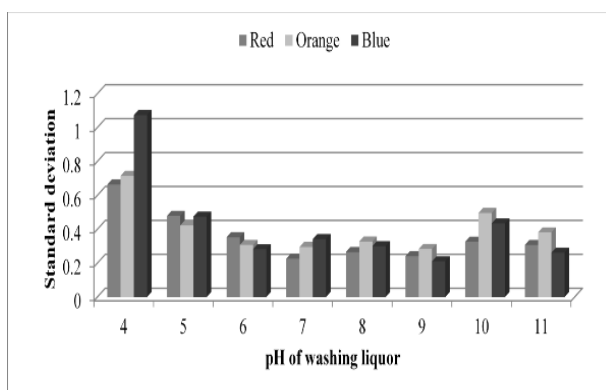


Fig. 4. Effect of pH of washing liquor on color uniformity of samples washed with copper salt

F. Fixation % and Color Change

1) Effect of Concentration:

While washing, huge amount of unfixed dye washed off which further resulted in a higher color change. In case of red dye, color change was observed that it was increasing with increase in concentration. At concentration of 1mg/l color change was near to 3, but at 5 mg/l it was more than 4. Besides, color change further continued to increase with the increasing concentration and at concentration of 20mg/l it was about 12. This change occurred not only due to the dye removal but also due to change in color components. Samples were converted to greener tone and the range of color change was out of acceptance. Similarly, for orange dye, higher color change was observed at 5mg/l and 10 mg/l concentration where the change was clearly visible. On the other hand, there was no sufficient effect of the salt concentration on color of blue dye. Concentration also had significant effects on fixation %. Hence, an increase in concentration resulted in the gradual decrease in fixation%. Lowest dye fixation of 68.8 was observed at maximum concentration of 20mg/l. On the other hand, the concentration had no effect on fixation of orange dye. Furthermore, gradual decrease was observed in fixation for blue dye up to 2mg/l and right after that it started increasing. Color change and fixation results were comparable to detergent and water wash, as it can be seen in Fig. 5.

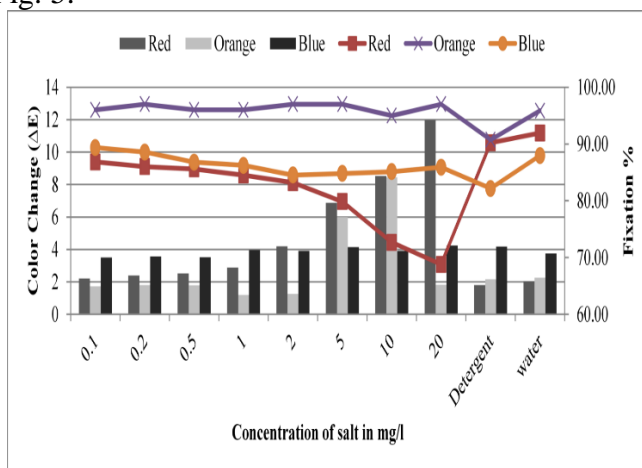


Fig. 5. Effect of salt concentration on color change and fixation% of samples washed with copper salt

2) Effect of Temperature:

Temperature had an expected effect on the color change and fixation in case of dichlorotriazine and vinylsulphone dyes. In this case, the color change tended to increase and fixation decreased with gradual increase in temperature, although this change was not so prominent. This can be attributed to continuing increase in complex formation ability of metal ions, higher water solubility, and increased hydrolysis of dye, with step wise increase in temperature. Furthermore, the highest color change and the lowest fixation percentage were obtained at 100°C. In case of monochlorotriazine dye, the highest color change was also obtained at 100°C, but on the other hand, there was no distinct effect on fixation%. Similarly, the maximum color change and the lowest fixation both were obtained at 100°C for vinylsulphone dye class. This can be mainly obtained because of dye class properties. The current study observed that only this dye exhibited poor stability and increased hydrolysis at higher temperature. Figure 6 comprises the color change and fixation % of the samples washed at different temperatures.

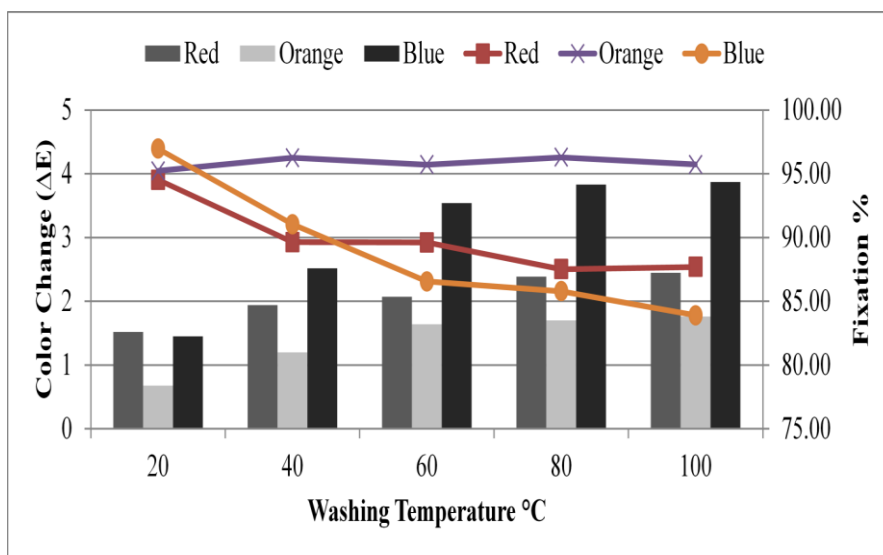


Fig. 6. Effect of washing temperature on color change and fixation% of samples washed with copper salt

3) Effect of Time

Time had minor effect on both the color uniformity and fixation in case of red dye. Anyhow, fixation percentage slightly decreased with the

increase in washing time and gradual increment in color change. This is due to the availability of longer reaction time. However, washing time has no prominent effect on both the color change and fixation of orange and blue dyes. It shows that washing of monochlorotriazine and vinylsulphone classes of dyes are independent of time. This can be seen in Figure 7 below.

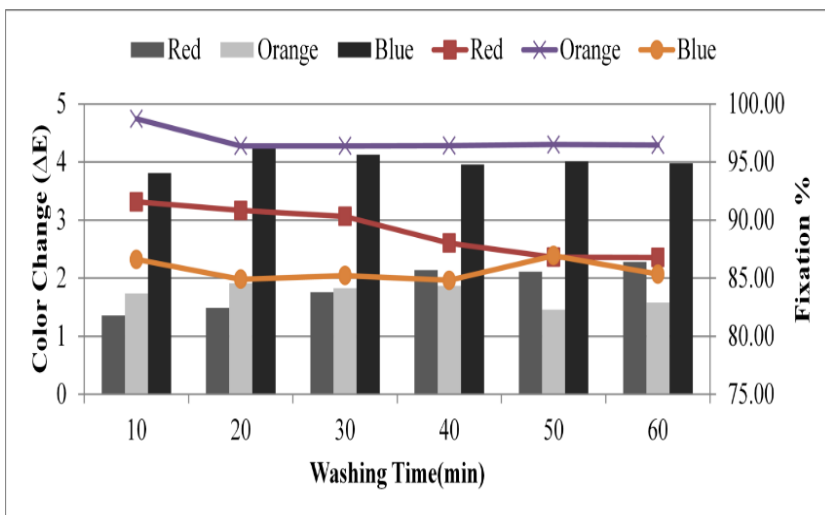


Fig. 7. Effect of washing time on color change and fixation% of samples washed with copper salt

4) Effect of pH:

The increase in pH level resulted in the increase in color change and the decrease in fixation percentage up to pH level 8 for red dye. Moreover, maximum color changes and lowest fixation were obtained at this level. Further increase resulted in the decrease in color change and increase in fixation percentage. This particular study expresses that dye and copper have maximum reactivity at this level. While for orange and blue dyes, alkaline pH levels were found to be more effective. In addition to it, the highest color change and the lowest fixation percentage were obtained at pH value of 11. This might be occurred because of the fact that the reactive dyes can be easily washed out at alkaline pH levels. Further, the reactive dyes are more prone to hydrolyze at these pH values as compared to the neutral pH values. Figure 8 represents the effect of pH of washing liquor on color change and fixation of reactive dyed fabric.

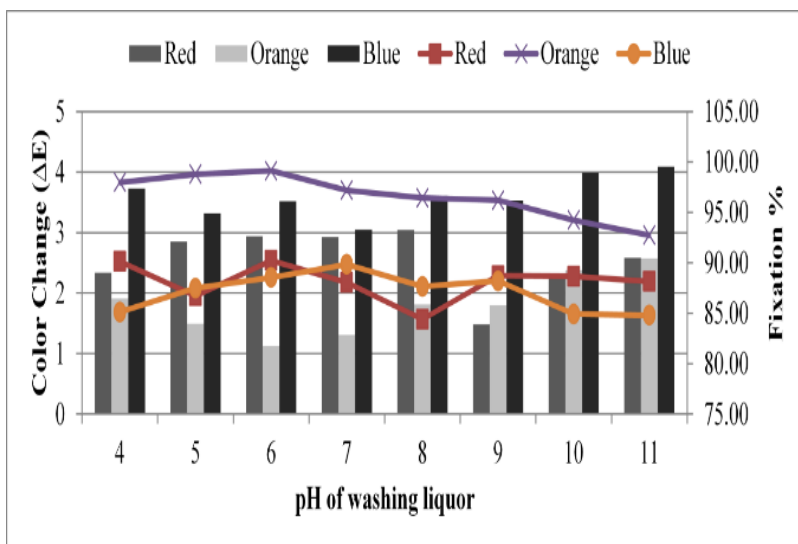


Fig. 8. Effect of pH of washing liquor on color change and fixation% of samples washed with copper salt

G. Washing Fastness

Although, the increase rate in color change of red and orange dyes was resulted due to the increase in concentration of copper salt. Furthermore, the color change rating tended to decrease gradually with the increase in concentration. Besides, in case of orange color, it was up to 6mg/l and after that it was increased again. This might be occurred because at the higher concentration, copper reacted vigorously with both the dyes.

This current study shows that both of these colors are sensitive to copper. However, it can also be attributed that monochlorotriazine and dichlorotriazine dyes are more prone to react with copper. Moreover, blue dye concentration has no effect on color change and staining. Hence, the stability of color or vinylsulphone's structure of dye against copper salt can be the main reason for no reaction of this color. Furthermore, temperature, time, and pH did not affect the color change and staining in case of red dye. This only happened because of the excellent washing fastness properties of dye. In addition to it, the change in temperature, time and pH also did not cause any color change for orange and blue dyes. However, resistance to staining was increased with the shifting of temperature from lower to higher levels and the change in time was found to be less effective. Both the dyes showed comparatively better ratings for staining at alkaline and acidic pH

levels than at neutral level. This is because at alkaline pH levels, reactive dyes hydrolyze quite easily and wash out with water, but at neutral levels they are stable. Hence, the washing fastness results of above-mentioned samples are shown in Table 1 below.

TABLE I
Washing Fastness of Samples Washed with Copper Metal Ions

Sr#	Factor	Color Change			Staining		
		Red	Orange	Blue	Red	Orange	Blue
1	Concentration	4~5	4~5	4~5	5	4~5	3~4
2	0.1,0.2,	4~5	4~5	4~5	5	4~5	3~4
3	0.5,1,2,	4	4~5	4~5	5	4~5	3~4
4	5,10,20	3~4	4	4~5	5	4~5	3~4
5	mg/l	3~4	3	4~5	5	4~5	3~4
6	Detergent,	3~4	2	4~5	5	4~5	3~4
7	Water	3	3	4~5	5	4~5	3~4
8	Respectively.	2~3	4~5	4~5	5	4~5	3~4
10		4~5	4~5	4~5	5	4~5	3~4
11	Temperature	4~5	4~5	4~5	5	3~4	2~3
12	20,40,	4~5	4~5	4~5	5	3~4	2~3
13	60,80,	4~5	4~5	4~5	5	4	3
14	100°C	4~5	4~5	4~5	5	4~5	3~4
15		4~5	4~5	4~5	5	4~5	3~4
16	Time	4~5	4~5	4~5	5	4	3~4
17	10,20,	4~5	4~5	4~5	5	4	3~4
18	30,40	4~5	4~5	4~5	5	4	3~4
19	50,60	4~5	4~5	4~5	5	4	3~4
20	Min	4~5	4~5	4~5	5	4~5	3~4
21		4~5	4~5	4~5	5	4~5	3~4

Sr#	Factor	Color Change			Staining		
		Red	Orange	Blue	Red	Orange	Blue
22	pH	4~5	4~5	4~5	5	4	3
23	4,5,6	4~5	4~5	4~5	5	4	3
24	7,8,9,	4~5	4~5	4~5	5	4	3
25	10,11	4~5	4~5	4~5	5	4	3
26		4~5	4~5	4~5	5	4	3
27		4~5	4~5	4~5	5	4~5	3
28		4~5	4~5	4~5	5	4~5	3~4
29		4~5	4~5	4~5	5	4~5	3~4

1. Rubbing Fastness:

Table 2, contains dry and wet rubbing fastness results of the samples. Metal ion concentrations were observed to be less effective on both dry and wet rubbing fastness. Similar results were obtained with detergent and water wash. Moreover, all dyes showed comparatively poor results only at 20°C. Further, time and pH did not cause any harm to rubbing fastness in rubbing fastness, samples are rubbed against bleached fabric. So, it mainly depends on the physical attachment of dye molecules which are present at the surface of the fabric with fiber or within fiber structure. However, the samples washed at 20°C showed poor results only because their washing at this temperature was not very effective. In addition to it, loosely attached dye molecules were still present on fabric surface. During rubbing fastness, these unfixed dye molecules present on the surface attached with white cloth, and gave poor rating of rubbing fastness.

TABLE II
Rubbing Fastness of Samples Washed with Copper Metal Ions

Sr#	Factor	Dry			Wet		
		Red	Orange	Blue	Red	Orange	Blue
1	Concentration	4~5	4		4 4~5	4	3~4
2	n	4~5	4		4 4~5	4	3~4

Sr#	Factor	Dry			Wet		
		Red	Orange	Blue	Red	Orange	Blue
3	0.1,0.2,	4~5	4	4	4~5	4	3~4
4	0.5,1,2,	4~5	4	4	4~5	4	3~4
5	5,10,20	4~5	4	4	4~5	4	3~4
6	mg/L	4~5	4~5	4	4~5	4	3~4
7	Detergent,	4~5	4~5	4	4~5	4	3~4
8	Water	4~5	4~5	4	4~5	4	3~4
9	Respectively.	4~5	4~5	4	4~5	4	3~4
10		4~5	4~5	4	4~5	4	3~4
11	Temperature	4~5	4	3~4	4~5	3~4	3
	20, 40,					4	
12	60, 80,	4~5	4~5	4	4~5	4	3~4
13	100 °C	4~5	4~5	4	4~5	4	3~4
14		4~5	4~5	4	4~5	4	3~4
15		4~5	4~5	4~5	4~5	4	4
16	Time	4~5	4~5	4	4~5	4	4
17	10,20,	4~5	4~5	4	4~5	4	4
18	30,40	4~5	4~5	4	4~5	4	4
19	50,60	5	4~5	4	4~5	4	4
20	Min	5	4~5	4~5	4~5	4	4~5
21		5	4~5	5	4~5	4	4~5
22	pH	5	4	5	4~5	4	4
23	4,5,6	5	4	4	4~5	4	4
24	7,8,9,	5	4	4	4~5	4	4
25	10,11	5	4	4	4~5	4	4

Sr#	Factor	Dry			Wet		
		Red	Orange	Blue	Red	Orange	Blue
26		5	4	4	4~5	4	4
27		5	4	4	4~5	4	4
28		5	4	4	4~5	4	4
29		5	4	5	4~5	4	4

III. Conclusion

The washing of textiles after dyeing is a least discussed topic as compared to other processes. In the current study, we focused on the effects of metal ions (cu) present in recycled water, after washing of the reactive dyed fabric. However, there were no prominent and uniform effects of copper on the color uniformity of dyed fabric. In any case, the presence of copper in water caused sufficient changes in color. It further produced unacceptable results because color change was observed along with an increase in the concentration for red and orange dyes. The color change that occurred was maximum for red (Dichlorotriazine), and orange (Monocholortriazine) was second in the list, but the effect was negligible on the blue dye (Vinylsulphone). Lower fixation and higher fastness ratings were obtained at higher temperatures. Moreover, the washing time and pH of washing liquor had no significant effect on the properties of fabric. For certain cases, washing in the presence of metal ions gave better results than washing in detergents and fresh water. So, the recycled water containing metal particles can be only used for washing after dyeing of the fabric instead of fresh water with detergent. Hence, only lower concentration of copper can be allowed in water for washing for reactive dyes. Consequently, the use of comparatively higher concentrations for certain classes of dyes would be possible.

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